

---

**(54) HIGH PURITY PHOSPHORIC ACID AND ITS PRODUCTION**

(11) 3-193614 (A) (43) 23.8.1991 (19) JP  
(21) Appl. No. 64-329462 (22) 21.12.1989  
(71) NIPPON CHEM IND CO LTD (72) YASUO YAMAZAKI(2)  
(51) Int. Cl<sup>5</sup>. C01B25/234

**PURPOSE:** To improve purity of phosphoric acid by dipping a specified crystallization tube in phosphoric acid to be a material, to precipitating phosphoric acid hemihydrate on the surface of the tube, taking out of the crystallization tube, and then allowing the hemihydrate to be sweated to remove impurities incorporated in the acid.

**CONSTITUTION:** The temperature of phosphoric acid to be the material having concn.  $\geq 70\text{wt.}\%$  (expressed by % hereinafter) is controlled at  $15-30^{\circ}\text{C}$ , and a crystallization tube of specified shape through which a coolant such as mixture solution of water/ethyleneglycol is made flow is dipped in the acid to precipitate phosphoric acid hemihydrate on the surface of the tube in a subcooling state at  $-2$  to  $-8^{\circ}\text{C}$ . Then the tube is taken out of the phosphoric acid liquid to separate the phosphoric acid hemihydrate from the mother liquid. The hemihydrate is made to be sweated while the fused amt. is controlled to  $10-40\text{wt.}\%$  to remove impurities incorporated in the inside/outside of the hemihydrate. Further, as necessary, the hemihydrate of phosphoric acid obtained by precipitation with super purity water or high purity phosphoric acid may be substituted and cleaned with super pure water of high purity phosphoric acid to remove impurities. Thus, the obtd. phosphoric acid is in high purity having impurities of  $\leq 25\text{ppb}$  Fe,  $\leq 3\text{ppb}$  Mn and  $\leq 40\text{ppb}$  Na expressed in terms of  $85\% \text{H}_3\text{PO}_4$  concn.

---

**(54) PRODUCTION OF TETRACALCIUM PHOSPHATE**

(11) 3-193615 (A) (43) 23.8.1991 (19) JP  
(21) Appl. No. 64-333471 (22) 22.12.1989  
(71) KYORITSU YOGYO GENRYO K.K. (72) YOSHIKO SUWA(1)  
(51) Int. Cl<sup>5</sup>. C01B25/32

**PURPOSE:** To increase single phase of phosphate by uniformly mixing low-crystallized hydroxyapatite and  $\text{CaCO}_3$  with the specified molar fraction of Ca to P and then sintering it.

**CONSTITUTION:** An aq. solution of  $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$  and aq. solution of  $(\text{NH}_4)_2\text{HPO}_4$  are compounded by the molar ratio of  $\text{Ca}/\text{P}=1.67$ , and pulverized in wet state to obtain low-crystalline hydroxy apatite. This apatite is then mixed and pulverized with  $\text{CaCO}_3$  by the molar ratio of  $\text{Ca}/\text{P}=2$  in wet or dried state to obtain a uniform powder as a material. This mixed powder is sintered at  $1350-1550^{\circ}\text{C}$  for 5-20 hours to obtain tetracalcium phosphate.

**KEMIRA OY**  
ESPOO RESEARCH CENTRE  
INFORMATION AND PATENT  
SERVICE